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ORGANOCHLORINES, PCB's AND CHLOROBENZENES IN CENTRIFUGED LAKE HURON WATER SAMPLES

M.A. Neilson
R.J.J. Stevens
J. Biberhofer

Water Quality Branch
Inland Water Directorate
Ontario Region

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The Lake Huron basin supports industrial and agricultural activities which contribute organic contaminants to the system, distributed so as to reflect sources and general usage patterns. The primary sources of organic contaminants to Lake Huron appear to be tributaries (Frank et al. 1981), point source discharges at the shoreline, and atmospheric deposition (Strachan and Huneault 1979). According to Eisenreich et al. (1980), estimated loadings of airborne contaminants to Lake Huron are second only to those of Lake Superior for all of the Great Lakes.

In their overview of existing contaminant data for Lake Huron, Kreis and Rice (1985) point out that historical studies on the various aspects of organic contamination show large discrepancies in yearly sampling coverage. The longest historical records, dating back to 1957, exist for water samples, however, sample size and detection limits hamper the utility of much of these data (Kreis and Rice 1985). For example, Glooschenko et al. (1976) collected 16-2L water samples in the Lake Huron-Georgian Bay system for analysis of PCB's, organochlorine (OC's) and organophosphorus pesticides, but all analyses were below the quantification limits. In contrast, Filkins and Smith (1982) collected approximately 100L of water at 18 sites in Lake Huron/Georgian Bay and analyzed for PCB's, pentachlorobenzene, hexachlorobenzene and DDT; quantifiable levels were found for every sample.

This report presents the concentrations of PCB's, organochlorines and chlorobenzenes found in 16 200-litre centrifuged surface (1 meter) water samples collected in the Lake Huron-Georgian Bay system. At each site the suspended sediment which resulted from centrifugation of 200L of water was insufficient for contaminant analysis. Implications of this sampling procedure are also briefly discussed.

METHODS

Samples were collected during April 30 - May 17, 1984 at the sites shown in Figure 1. Water was delivered, through a Nalgene 80/30 hose using a March magnetic-drive submersible pump, to a Westfalia continuous flow centrifuge at a rate of 6 litres/minute. The effluent was immediately extracted in the field using the APLE system (McCrea and Fischer 1985). Extracts were then returned to the National Water Quality Laboratory in Burlington, Ontario for clean-up, fractionation, identification and quantification (Environment Canada 1979). Analyses were performed for polychlorinated biphenols (PCB's), identified against a standard containing Aroclors 1242, 1254 and 1260; hexachlorocyclohexane isomers, α-and γ-BHC (lindane); heptachlor and heptachlor epoxide; aldrin; dieldrin; endrin; cis-(α -) and trans-(γ -) chlordane; α- and β-endosulfan; mirex; methoxychlor; DDT and it's derivatives; trichlorobenzenes (1,3,5-, 1,2,4- and 1,2,3-TCB); tetrachlorobenzenes (1,2,4,5-, 1,2,3,5- and 1,2,3,4-TeCB); pentachlorobenzene (QCB) and hexachlorobenzene (HCB). Analytical techniques were insufficient for accurately measuring the lower molecular weight mono- and dichlorobenzenes although the latter were included for comparison with existing literature values.

Detection limits, effectively reduced by a factor of 200 due to the volume of sample collected, were ~ 0.002 ng.L^{-1} for organochlorines, ~ 0.025 ng.L^{-1} for dichlorobenzenes, ~ 0.004 ng.L^{-1} for tri-, tetra- and pentachlorobenzenes and ~ 0.05 for PCB's.

Two 4L methylene chloride blanks, representing that volume of solvent used to extract the samples, plus two 4L OC and PCB spikes were submitted with the samples as quality control checks.

RESULTS AND DISCUSSION

Table 1 lists the results from the quality control studies. Both blanks were found to have OC and PCB concentrations below the limit of detection (0.4 and 9.0 $ng.L^{1}$, respectively) although one 4-litre blank did show an instrument response of 19.0 ng PCB's ($\sim$ 4.7 $ng.L^{-1}$). Ortho-dichlorobenzene was found in both blanks (1.1 and 7.7 $ng.L^{-1}$), while meta- and para- dichlorobenezene were only detected in the first blank (at $\sim$ 1.2 and 2.4 $ng.L^{-1}$, respectively).

Recoveries for the 2 spikes varied between 24 and 232% although, for 8 of the 19 compounds, recoveries for both spikes were of the order of 90-100%. Extremely low recoveries, however, were found for alpha-endosulfan (28% on both spikes), beta-endosulfan (30 and 32%) and PCB's (31% on both spikes). Conversely, high recoveries were found for aldrin (232 and 152%) and p,p'-DDE (229 and 125%), while heptachlor recoveries for the two spikes were inconsistent (146 and 24%). Heptachlor and aldrin, however, were not expected to be found in ambient water and p,p'-DDE was rarely detected.

Table 2 lists, for each site, the results of all analyses performed with the range of concentrations observed for each compound. Aldrin, heptachlor, DDT and its derivatives (except DDE), mirex, methoxychlor, m-DCB, (1,3,5-) TCB, (1,2,4,5-) and (1,2,3,5-) TeCB were below the quantifiable levels in all samples collected. Alpha-endosulfan and DDE were detected at 4 and 2 sites, respectively, while the remaining 16 contaminants were ubiquitous in their distribution. Figures 1 through 16 plot the observed concentrations for each of these latter contaminants.

I. POLYCHLORINATED BIPHENYLS

PCB's were found in surprisingly low levels in this study ($<$ 0.4 $ng.L^{-1}$) relative to the findings of Filkins and Smith (1982) who, for the same lake, reported PCB concentrations in the range of

0.135-3.232 $ng.L^{-1}$ during 1980-81. As previously discussed, our spike recoveries for PCB's were only 31%, however, adjusting our results accordingly was still insufficient to attain these levels. The work of Filkins and Smith is itself suspect as PCB concentrations at the same site showed more than a 12-fold difference between the 2 years (1.805 and 0.135 $ng.L^{-1}$ for 1980 and 1981, respectively). Yet, Chan's (1986) Lake Superior data showed PCB's in the 1-5 $ng.L^{-1}$ range reaffirming that our values were low.

DiToro et al. (1982) studied the partitioning of PCB's between the dissolved and particulate phases of Saginaw Bay samples and found the concentration of suspended adsorbent to affect the partition coefficient. The coefficient consistently decreased as adsorbent concentration increased. This is of considerable consequence to this study, as our Lake Huron-Georgian Bay sites were located in the open waters where suspended sediment load is extremely low. Thus, the partition coefficient would be correspondingly high with the possible result that, due to centrifuging, the PCB's were removed from our samples.

II. ORGANOCHLORINES

As in the rest of the Great Lakes Basin (McCrea et al. 1985; Biberhofer and Stevens 1986; Chan 1986), of all the organochlorines, α -and γ-BHC were found in the highest concentrations in Lake Huron. (α:γ) ratios for Lake Huron-Georgian Bay were consistently in the range of 14.5-15.2, except at stations LH69 and LH71 (at the St. Mary's River outflow to Lake Superior) where (α : γ) ratios were 10.02 and 10.45, respectively, and in Collingwood Harbour (=7.02). This isomer ratio can be used to characterize different water masses, a concept employed by Biberhofer and Stevens (1986) to delineate sources.

Since direct precipitation over the lake accounts for 24% of the Lake Huron-Georgian Bay water budget, (Upper Lakes Reference Group 1977), this mode is a likely source of such substances. For example, assuming uniform precipitation concentrations of 13.3 and 6.0 $ng.L^{-1}$ for α-BHC and lindane, respectively (Strachan and Huneault 1979), and annual mean rainfall to the lake surface of 171 cm (C.H. Chan, pers. comm.), this would result in loadings of approximately 1356 and 612 kg per annum.

Although aldrin was not detected at any of the stations, the end-product of its biotransformation, dieldrin, was consistently found, generally at levels one-third that of lindane (ie., 0.197 - 0.366 $ng.L^{-1}$). The presence of dieldrin in the Great Lakes Basin is a result of its widespread use in North America prior to 1970. Concentrations observed in Lake Huron were similar to those reported by McCrea et al. (1985) and Biberhofer and Stevens (1986) for whole water samples collected in the Lower Great Lakes. Because dieldrin is soluble in water (200 ppb at 25°C, Weil et al. 1974), the impact of centrifugation would be minimal. According to the objectives outlined in the 1978 Canada-US Great Lakes Water Quality Agreement, the sum of the concentrations of aldrin and dieldrin must not exceed 1.0 $ng.L^{-1}$, hence, Lake Huron appears to be in compliance.

Several studies have shown that heptachlor can be biotransformed by oxidation to heptachlor epoxide (US EPA 1979). As above, the parent compound was not detected at any of the stations, while heptachlor epoxide was widespread (0.071-0.211 $ng.L^{-1}$). There were no incidences of objective non-compliance as the sum of heptachlor and its metabolic product was always less than 1.0 $ng.L^{-1}$.

Continuing with the organochlorine found in the next highest concentration, endrin was detected at every station, except at the Lake Huron-Saginaw Bay interface (LH95) and in southern Lake Huron (LH01), consistently at levels 1.5-2 orders of magnitude below the 2.0 $ng.L^{-1}$ objective. Although Chan (1986) reported endrin to be detected at only

35% of the 20 sites sampled in Lake Superior, concentrations were of the same order of magnitude as those found in Lake Huron during this study. Lake Ontario whole water samples, however, contained at least twice as much endrin (Biberhofer and Stevens 1986). It is unlikely that centrifugation of Lake Huron water was responsible for the difference observed relative to Lake Ontario, as endrin is most abundant in the aqueous phase (McCrea et al. 1985).

Both cis-(α-) and trans-(γ -) chlordane were frequently detected (at 10 and 14 stations, respectively), and although concentrations in the centrifuged water were similar for the two isomers, they were often only 2 to 3 times the quantifiable level, never approaching the objective of 60 $ng.L^{-1}$. Oloffs et al. (1972;1973) and Oloffs and Albright (1974) conducted experiments in which cis- and trans-chlordane were placed in flasks containing natural water and sediments. When sediment was present, more than 80% of the chlordane was recovered from the sediment. This contrasts with the findings of McCrea et al. (1985) who reported trans-chlordane to be found exclusively in the aqueous phase in Lake Erie and Niagara River, while the partitioning of the cis-isomer was variable. It is, therefore, possible that chlordane was removed from the water samples through centrifugation.

Endosulfans were found at concentrations similar to chlordanes although, as mentioned earlier, α -endosulfan was only found at 4 sites: at the 2 stations at the outflow of Lake Superior via St. Mary's River, and the 2 Georgian Bay nearshore stations. It is interesting to note that, while -endosulfan was reported at every station (0.014-0.035 $ng.L^{-1}$), the 2 Lake Superior stations were only $\sim$50% of the mean concentration. Greve and Wit (1971) found endosulfan to be more than 75% associated with particulate matter, again suggesting that centrifugation may have removed significant quantities of this substance. Concentrations observed in Lake Superior whole water samples (Chan 1986) were as much as an order of magnitude greater than Lake Huron centrifuged samples, and the α isomer was the more dominant.

With the exception of Georgian Bay stations 27 and 42, where pp'-DDE was reported at concentrations of 0.016 and 0.012 $ng.L^{-1}$, respectively, DDT and its metabolites (= DDT-R) were not found. In a 1980-81 study of the offshore waters of Lake Huron-Georgian Bay, Filkins and Smith (1982) reported pp'-DDE and -DDT in all twenty whole water samples, at concentrations ranging from 0.006 to 0.090 $ng.L^{-1}$ and 0.007 to 0.062 $ng.L^{-1}$, respectively. Biberhofer and Stevens (1986) found DDT-R to be ubiquitous in Lake Ontario, generally in the 0.1 - 0.2 $ng.L^{-1}$ range. DDT-R was also widespread in Lake Superior, mainly as p,p'-DDE and -DDT, with concentrations ranging from 0.016-0.661 $ng.L^{-1}$ in whole water samples (Chan 1986). As sorption of DDT-R to suspended and bottom sediments is an important process (Environmental Protection Agency 1979), perhaps the DDT-R thus far identified in the Great Lakes Basin is strictly associated with the sediment and, thus, by centrifuging our samples, the DDT-R was extracted.

It is also possible that, without centrifuging, 1984 DDT-R levels in Lake Huron/Georgian Bay would have been below the quantifiable level ($< .002\ ng.L^{-1}$). DDT-R was first detected in Lake Huron in 1974, when concentrations in excess of the 3.0 $ng.L^{-1}$ Agreement objective were reported for Thunder and Saginaw Bays (Kreis and Rice 1985). Concentrations in the open lake have since decreased by one, then 2 orders of magnitude through 1981. Similarly, DDT-R concentrations in fish (Fornay 1982) and gull eggs (Weseloh et al. 1979) have shown marked reductions throughout 1973-1980. For example, DDT-R levels in gull eggs at Chantry and Double Islands decreased to 0.06 $ug.g^{-1}$ in 1979 (Great Lakes Water Quality Board 1981) from 21.9 and 14.9 $ug.g^{-1}$, respectively, in 1974.

III. CHLOROBENZENES

As stated earlier, only p- and o-DCB, (1,2,4-) and (1,2,3-)TCB, (1,2,3,4-)TeCB, QCB and HCB were detected in any of the Lake Huron samples. With the exception of the two Lake Superior outflow stations

(LH71, LH69), which showed an almost total absence of chlorobenzenes (Σ CB = 0.012, 0.032 $ng.L^{-1}$, respectively), these CB's were ubiquitous in distribution.

Oliver and Nicol (1982) were not able to find o-DCB in any of their Lake Huron water samples but reported p-DCB at concentrations of 3 to 6 $ng.L^{-1}$. The results of our study showed o-DCB to be of the same concentration range as p-DCB (0.056-0.536 $ng.L^{-1}$). It has been suggested that volatilization of the lower order CB's occurs during rotoevaporation at the concentration stage of the analytical procedure used for our study. Biberhofer and Stevens (1986), who also employed rotoevaporation, were unable to detect dichlorobenzenes in Lake Ontario, while Oliver and Nicol (1982) found both o- and p-DCB (Table 3). An apparent discrepancy arises, however, if it is assumed that o- and p-DCB are present in Lake Huron in similar quantities, as our data suggests, and that both DCB's are equally volatilized by rotoevaporation of the samples (vapour pressures at 25°C for o- and p-DCB are 1.5 and 1.2 Torr, respectively). Why did Oliver and Nicol (1982) then not find o-DCB?

A further indication that considerable quantities of CB's were lacking from our data is that Oliver and Nicol (1982) found HCB to comprise $< 0.5\%$ of the total CB's (dichloro- to hexachloro-) in lake water, while our results averaged 9%.

The log octanol/water partition coefficients (log P) for chlorobenzenes (log $P_{DCB} \sim 3.38$, log $P_{TCB} \sim 4.26$, log $P_{HCB} = 6.18$) indicate that sorption may be an important process for this class of compounds. Centrifugation of our samples is then another possible cause for the lower CB levels observed in our samples.

SUMMARY AND RECOMMENDATIONS

Aldrin, heptachlor, DDT and its metabolites, mirex, methoxychlor, m-DCB, (1,3,5-)TCB, (1,2,4,5-) and (1,2,3,5-)TeCB were below the quantifiable level in all samples collected. Alpha-endosulfan and p,p'-DDE were detected at 4 and 2 sites, respectively, while the remaining 16 contaminants were ubiquitous in distribution. The concentration ranges of organochlorines present in Lake Huron centrifuged water samples were, in decreasing order, α-BHC (4.320 - 12.190 $ng.L^{-1}$), γ-BHC (0.475 - 0.835 $ng.L^{-1}$), dieldrin (0.197 - 0.366 $ng.L^{-1}$), heptachlor epoxide (0.071 - 0.211 $ng.L^{-1}$), endrin ($<$ 0.002 - 0.052 $ng.L^{-1}$), β-endosulfan (0.014 - 0.035 $ng.L^{-1}$) and chlordane ($<$0.002 - 0.032 $ng.L^{-1}$).

DiToro et al. (1982) found the concentration of suspended adsorbent to have an inverse effect on the partition coefficient of PCB's, indicating that, for our open lake study, PCB's would selectively partition with the suspended sediment phase. This then implies that centrifugation would result in lower PCB levels as was observed in our samples. A similar explanation has been suggested to rationalize chlordane, endosulfan, DDT-R and chlorobenzene concentrations reported herein which appear to be lower than expected when compared to current literature values. It is, therefore, recommended that whole lake samples be collected in the future, with a few centrifuged (plus resultant suspended sediment) samples for comparative purposes.

One further recommendation is that all samples be spiked with at least a pair of organic contaminants (at least one of a volatile nature) not expected to be found in the samples. Recently the NWQL tested this protocol using δ- and β-BHC and etnylan on Lake Ontario water samples, however, the latter was not successfully extracted from the samples.

The NWQL has begun using a modified analytical procedure for concentration of sample extracts that will eliminate the problem of volatilization which purportedly resulted from rotoevaporation. It is thus expected that chlorobenzene recoveries will improve. Bromobenzene, or another compound of similar molecular weight, however, should be added to quality control (spike) samples to allow quantification of CB recoveries.

Furthermore, in light of the paucity of historical data on organic contaminants in the water phase, such work should be continued to establish a substantial database.

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TABLE 1.

QUALITY CONTROL SAMPLE RESULTS FOR ORGANOCHLORINES, PCB's AND CB's

	BLANKS (ng)		SPIKE RECOVERY (%)	
α-BHC	0.7	ND	100	96
γ-BHC	ND[a]	ND	76	78
heptachlor	ND	ND	146	24
heptachlor epoxide	ND	ND	92	102
aldrin	ND	ND	232	158
dieldrin	ND	ND	99	101
cis-chlordane	ND	ND	93	93
trans-chlordane	ND	ND	90	92
α-endosulfan	ND	ND	28	28
β-endosulfan	ND	ND	30	32
endrin	ND	ND	96	108
pp'-DDE	ND	ND	229	125
op-DDT	ND	ND	77	102
pp'-TDE	ND	ND	87	95
pp'-DDT	ND	ND	100	67
mirex	ND	ND	124	108
methoxychlor	ND	ND	96	75
PCB's	ND	19.01	31	31
m-DCB	4.7	ND	-[b]	-
p-DCB	9.6	ND	-	-
o-DCB	4.4	30.6	-	-
Σ TCB	ND	ND	-	-
Σ TeCB	ND	ND	-	-
QCB	ND	ND	-	-
HCB	ND	ND	54	100

[a]ND = not detected
[b] There were no spikes added for any CB's except hexa-.

Table 2. Organochlorine, PCB and CB results for each site sampled and associated detection limits (dl). All values are in $ng.L^{-1}$

	dl	LH71	LH69	LH76	LH77	LH84	LH65	LH63	LH33	LH29	LH95	LH09	LH01	GB42	GB27	GB29	GB09
a-BHC	0.002	6.585	7.065	11.640	7.845	8.470	8.910	6.725	10.305	8.840	9.595	9.895	10.375	11.535	8.595	12.190	4.320
g-BHC	0.002	.630	.705	.805	.520	.590	.615	.475	.700	.582	.680	.670	.690	.785	.595	.835	.615
heptachlor	0.002	ND[1]	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
heptachlor epoxide	0.002	.098	.085	.211	.086	.119	.205	.156	.165	.153	.206	.191	.134	.173	.071	.208	.082
aldrin	0.002	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
dieldrin	0.002	.197	.232	.321	.202	.219	.325	.278	.261	.294	.297	.366	.332	.266	.210	.313	.210
cis-chlordane	0.002	ND	ND	.014	.009	ND	.008	ND	.011	.011	.018	ND	.015	.004	.006	.004	ND
trans-chlordane	0.002	.005	.005	.006	.003	ND	.004	ND	.007	.021	.004	.006	.015	.004	.004	.003	.004
a-endosulfan	0.002	.010	.013	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	.009	ND	.011
b-endosulfan	0.002	.014	.017	.031	.019	.026	.035	.026	.024	.033	.028	.031	.023	.035	.021	.031	.026
endrin	0.002	.048	.042	.048	.030	.034	.052	.034	.048	.051	ND	.045	ND	.040	.026	.044	.023
p,p'-DDE	0.002	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	.012	.016	ND	ND
o,p-DDT	0.002	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
p,p'-TDE	0.002	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
p,p'-DDT	0.002	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
mirex	0.002	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
methoxychlor	0.002	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
PCB's	0.05	.112	.170	.126	.135	.257	.105	.153	.168	.209	.175	.394	.319	.158	.166	.253	.076
m-DCB	0.025	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
p-DCB	0.025	ND	.026	ND	ND	.505	.505	.547	.026	.060	.325	.056	(.146)	.386	ND	.470	.265
o-DCB	0.025	ND	ND	ND	ND	.248	.210	.226	.056	.287	.255	.149	.078	.535	ND	.187	.160
1,3,5 - TCB	0.004	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2,4 - TCB	0.004	.009	ND	.058	.055	.170	.161	.151	.077	.187	.185	.148	.181	.058	.051	.177	.066
1,2,3 - TCB	0.004	ND	ND	.011	.008	.017	.018	.017	.015	.022	.022	.021	ND	.017	.010	.020	.011
1,2,4,5 - TeCB	0.004	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2,3,5 - TeCB	0.004	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2,3,4 - TeCB	0.004	ND	ND	.022	.021	.024	.024	.018	.017	.029	.030	.031	.027	.023	.016	.027	.014
OCB	0.004	ND	ND	.009	.009	.009	.008	.009	.006	.012	.010	.014	.014	.009	.011	.012	.006
HCB	0.002	.003	.006	.022	.022	.024	.023	.025	.023	.032	.022	.030	.030	.018	.021	.019	.012

[1]ND = Not detected.

TABLE 3.

CHLOROBENZENE CONCENTRATIONS IN LAKES HURON AND ONTARIO ($ng.L^{-1}$)

-	Huron	Huron (a)	Huron (c)	Ontario (a)	Ontario (b)
o-DCB	.025-.535	1.0		2-7	0.1
m-DCB	.025	1.0		1.0	0.1
p-DCB	.025-.547	3-6		33-64	0.1
1,3,5-TCB	.004	0.1		0.1-0.4	0.02-0.08
1,2,4-TCB	.004-.187	0.1-0.4		0.3-1	0.02-1.36
1,2,3-TCB	.008-.022	0.1		0.1-0.2	0.02-0.67
1,2,4,5-TeCB	.004	0.05		0.05-0.3	0.02-0.32
1,2,3,5-TeCB	.004	0.05		0.05	
1,2,3,4-TeCB	.004-.031	0.04-0.06		0.05-0.5	0.02-0.57
QCB	.004-.014	0.03-0.04	0.001-0.007	0.01-0.6	0.02-0.22
HCB	.003-.032	0.02-0.1	0.002-0.019	0.02-0.1	0.02-0.10

(a) Oliver and Nicol (1982)

(b) Biberhofer and Stevens (1986)

(c) Filkins and Smith (1982)